

CLAIMS

1. A compound which is a crystalline Form X of nateglinide.
2. The compound of claim 1, having an X-ray diffraction pattern, expressed in terms of 2 theta angles, that includes five or more peaks selected from the group consisting of 3.95 ± 0.09 , 4.89 ± 0.09 , 5.18 ± 0.09 , 6.78 ± 0.09 , 7.79 ± 0.09 , 10.32 ± 0.09 , 13.51 ± 0.09 , 14.00 ± 0.09 , 16.98 ± 0.09 , 17.94 ± 0.09 , 18.85 ± 0.09 , 19.17 ± 0.09 , 20.32 ± 0.09 , 21.12 ± 0.09 , 22.52 ± 0.09 , 23.76 ± 0.09 , 24.46 ± 0.09 , 27.36 ± 0.09 , 28.17 ± 0.09 , 30.88 ± 0.09 , 31.25 ± 0.09 , 32.61 ± 0.09 , and 41.65 ± 0.09 degrees.
3. The compound of claim 2, wherein said X-ray diffraction pattern includes at least the peaks at 3.95 ± 0.09 , 14.00 ± 0.09 , and 16.98 ± 0.09 degrees.
4. The compound of claim 2, wherein said X ray diffraction pattern includes peaks at 3.952, 14.039, 16.98, 20.325, 21.120, 17.942, 6.776, 13.515, and 18.853 degrees.
5. The compound of claim 1, having an infrared absorption spectrum with absorption bands at about 3353 cm^{-1} , about 2937 cm^{-1} , about 2868 cm^{-1} , about 1743 cm^{-1} , about 1646 cm^{-1} , about 1597 cm^{-1} , about 1541 cm^{-1} , about 1445 cm^{-1} , about 1208 cm^{-1} , about 1190 cm^{-1} , about 1110 cm^{-1} , about 697 cm^{-1} , and about 607 cm^{-1} .
6. The compound of claim 1, having substantially the same X-ray diffraction pattern as that shown in Figure 1.
7. The compound of claim 6, having substantially the same infrared spectrum as that shown in Figure 2.
8. A composition comprising nateglinide as a solid, wherein at least 80% by weight of said solid nateglinide is its crystalline Form X having an X-ray diffraction pattern, expressed in terms of 2 theta angles, that includes five or more peaks selected from the group consisting of 3.95 ± 0.09 , 4.89 ± 0.09 , 5.18 ± 0.09 , 6.78 ± 0.09 , 7.79 ± 0.09 , 10.32 ± 0.09 , 13.51 ± 0.09 , 14.04 ± 0.09 , 16.98 ± 0.09 , 17.94 ± 0.09 , 18.85 ± 0.09 , 19.17 ± 0.09 , 20.32 ± 0.09 , 21.12 ± 0.09 , 22.52 ± 0.09 , 23.76 ± 0.09 , 24.46 ± 0.09 , 27.36 ± 0.09 , 28.17 ± 0.09 , 30.88 ± 0.09 , 31.25 ± 0.09 , 32.61 ± 0.09 , and 41.65 ± 0.09 .
9. The composition of claim 8, wherein said X-ray diffraction pattern includes at least the peaks at 3.95 ± 0.09 , 14.00 ± 0.09 , and 16.98 ± 0.09 degrees.
10. The composition of claim 8, wherein at least 90% by weight of said solid nateglinide is its crystalline Form X.
11. The composition of claim 8, wherein at least 95% by weight of said solid nateglinide is its crystalline Form X.

12. The composition of claim 8, wherein at least 99% by weight of said solid nateglinide is its crystalline Form X.
13. The composition of claim 8, wherein said solid nateglinide is substantially free of its crystalline Forms H and B.
14. The composition of claim 8, wherein at least 1% of said solid nateglinide is not its crystalline Form X.
15. The composition of claim 8, wherein at least 5% of said solid nateglinide is not its crystalline Form X.
16. A pharmaceutical composition comprising the compound of claim 1 and a pharmaceutically acceptable carrier or diluent.
17. The pharmaceutical composition of claim 16, further comprising one or more pharmaceutically acceptable excipients.
18. The pharmaceutical composition of claim 17, which is a solid dosage form for oral administration.
19. The pharmaceutical composition of claim 18, wherein said solid dosage form is a tablet.
20. A process for preparation a crystalline form X of nateglinide, said process comprising:
 - a. providing a solution of nateglinide in an aromatic hydrocarbon solvent;
 - b. cooling the solution until a precipitate is formed; and
 - c. isolating the precipitate, which is the crystalline form X of nateglinide.
21. The process of claim 20, further comprising drying the isolated precipitate.
22. The process of claim 20, wherein said aromatic hydrocarbon solvent is selected from the group consisting of benzene, ethyl benzene, toluene, and xylene.
23. The process of claim 20, wherein said aromatic hydrocarbon solvent is xylene or ortho-xylene.
24. The process of claim 20, wherein the starting nateglinide is crystalline Form H, crystalline Form B, or a mixture thereof.
25. The process of claim 20, wherein said providing step includes heating a mixture of the starting nateglinide and the aromatic hydrocarbon solvent to a temperature of from about 40°C to about 130°C until the solution is formed.

26. The process of claim 25, wherein the mixture is heated to from about 60°C to about 70°C.
27. The process of claim 20, further comprising filtering said provided solution of nateglinide prior to said cooling step.
28. The process of claim 20, wherein the solution of nateglinide is cooled to from about 25°C to about 35°C.
29. A process for making crystalline form X of nateglinide, said process comprising:
- a. forming a solution of nateglinide in xylene or ortho-xylene at from about 50°C to about 70°C;
 - b. cooling the solution from 25°C to about 35°C to form a precipitate;
- and
- c. filtering said precipitate.
30. The process of claim 9, further comprising drying the precipitate.
31. A compound produced by the process of claim 20.
32. A compound produced by the process of claim 29.
33. A compound produced by the process of claim 30.